Synopsis on

"SYNTHESIS, CHARACTERIZATION AND GAS SENSING APPLICATIONS OF 2D DICHALCOGENIDE (MXene)."

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PHYSICS

By

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Abstract of Proposed Work Plan/Problem

Synthesis, Characterization and gas sensing applications of 2D Dichalcogenide (MXene).

1. Introduction

Since the exfoliation of graphene, a significant amount of research has expanded to other 2D layered materials, due to their extraordinary electronic, mechanical and optical properties. Beyond graphene, transition metals dicalcogenides , phosphorene and their derivatives are examples of the most investigated 2D materials. Recently, the discovery of a 2Dlayered compounds family, namely "MXenes" (1). In materials science, MXenes are a class of two-dimensional inorganic compounds , that consist of atomically thin layers of transition metal carbides, nitrides, or carbonitrides. (2).

Transition Metal Chalcogenides (TMCs) have been attracted a lot of attention because of their unique electronic and optical properties. TMCs have shown promising approaches towards phototransistors and other optoelectronic devices. Two-dimensional (2D) transition metal nitrides, carbonitrides , carbides are the newest material families which are most versatile in nature. An elegant exfoliation technique has been successfully employed to synthesize the family of 2D transition metal nitrides, carbonitrides, and carbides, which are also called as MXene, from layered MAX phases in recent years (3). MXenes are the most recent and emerging class of 2D materials that have aroused worldwide interest owing to their structural similarity with graphene (4). Mxenes, transition metal carbides, nitrides and carbonitrides, are generally described by a formula Mn + 1XnTx, where M represents a transition metal (Ti, V, Cr, Mo, Nb, etc.), X stands for C or N atom and T is a terminal group which can be O, OH, F, Cl or S (5). The high electrical conductivity of MXenes,

diverse stacking structures and controllable surface terminations also provide an intriguing pathway to modify materials structure, properties and sensing performance (6).

MXenes is expected to go beyond the boundaries of existing sensor technology and can be used as an alternative sensor material. Therefore, it is necessary to comprehensively introduce the wide application of MXenes in the sensing field, promote the development of MXenes in the sensing field, and realize low-cost, high-performance, early large-scale application and industrialization. (7)

2. Literature Review and Development in the Subject (previous work done in the relevant area):

a) The first computational study to demonstrate the potential of MXenes as a gas sensor or capturer was conducted by **Yu et al.** (8)

b) ***Zhang et al*** developed composite hydrogel based on Ti3C2- Tx MXene and polyvinylalcohol (PVA) as a strain sensor. The fabricated PVA/Ti3C2Tx MXene composite hydrogel displayed excellent tensile strain sensitivity with a GF of 25 which is about ten times greater than that of a pristine hydrogel. In particular, the PVA/Ti3- C2Tx MXene composite hydrogel demonstrated much higher sensitivity with regards to compressive strain (GF = 80) than under tensile strain. (9)

c) **Guo et al.** studied the VOC sensing performance of Mo_2CT_x MXene sensors. By selective etching of Ga layers from the Mo_2Ga_2C precursor using HF, multilayer Mo_2CT_x MXenes

were produced. The concentration of the MXene (0.066 mg/mL) and the sonication time (8 h) were optimized in order to achieve a higher VOCs sensitivity. The optimized Mo_2CT_x MXene sensor offered an excellent selectivity toward toluene against the other VOCs with sensitivity of 0.0366 Ω/ppm at 140 ppm and a LOD of 220 ppb. (10)

d) **Cho et al.** also reported the synthesis of two phases of pure molybdenum carbide materials (α-MoC_{1-x} and β-Mo_2C) with structures similar to MXenes (without the expression T_x in the chemical formula) via a temperature-programmed reduction technique. While both phases show metallic behavior, α-MoC_{1-x} MXene (200–400 ohm) has higher resistance compared to β-Mo_2C (tens of ohm). Due to the excellent catalytic properties and high electrical conductivity, the molybdenum carbide-based sensors showed great sensing performance as well as ultrahigh SNR. The sensors were exposed to various gases like acetone, ethanol, propanal, hexane, toluene, NO_2, SO_2, and NH_3. (10)

e) **Sinha et al**. presented a review which discusses the latest advances on the applications of MXene for sensing at that time and highlights their important role in analytical sensing, including electrochemical, gas adsorptive, wearable sensors and photoluminescent sensors as well. **Kalambate et al**. published a review which introduces the latest development of electrochemical (bio) sensors, including biomarkers, drug and environmental pollution sensors. (7)

f) Very recently, an informative review by **Xin et al.** gives an idea about the applications of MXenes wearable sensors . (7)

g) **Gogotsi** said. “MXene can detect gases in the 50-100 parts per billion ranges, which is below the concentration necessary for current sensors to detect diabetes and a number of other health conditions.” (11)

h) **Yuan et al**. developed a three-dimensional (3D) MXene framework through combined electrospinning and selfassembly approach for the detection of volatile organic compounds. (12)

3. Objective of research/Proposed Hypothesis:

To syntheses a MXene material by one of the present synthesizing method and check the capabilities of that material towords Gas Sensors and check signal to noise ratio.

Therefore, present objective of the work is to study the MXene based Gas Sensors and determine the following.

- sensitivity
- selectivity
- response and recovery time
- detection limit and resolution
- stability and working temperature
- Signal to noise ratio.
    - *“signal-to-noise” ratio in the world of chemical sensors : it is used to rank the quality of sensors — picking up more signal and less noise is the goal.* (13)

4. Methodology to be adopted:

The two major difficulties encountered in the synthesis of nitride-based MXenes are higher formation energy of Mn+1ANn and compromised the stability of the Mn+1Nn layers employed in the etchant (conventionally HF). Soundiraraju et al. addressed this problem through the synthesis of Titanium Nitride (Ti2N). (14) So here we are using carbide-based MXene.

Fabrication routes of MXenes are primarily focused on the removal of the more reactive “A” atoms from Mn+1Xn layers without any structural alterations. This feature is attributed to the relative weakness of M-A bonds compared to the M-X bonds, which is a stronger metallic covalent bonds. MAX phases are tightly wired to each other by strong van der Waals interaction, which complicates exfoliation more than similar 2D materials like Graphene, Phosphorene, etc. Literature suggests that around twenty MXenes are successfully produced to date from more than 70 types of MAX phases. The dissolving and etching of the A layers require aggressive acidic conditions, and hydrofluoric acid (HF) is conventionally employed (15).

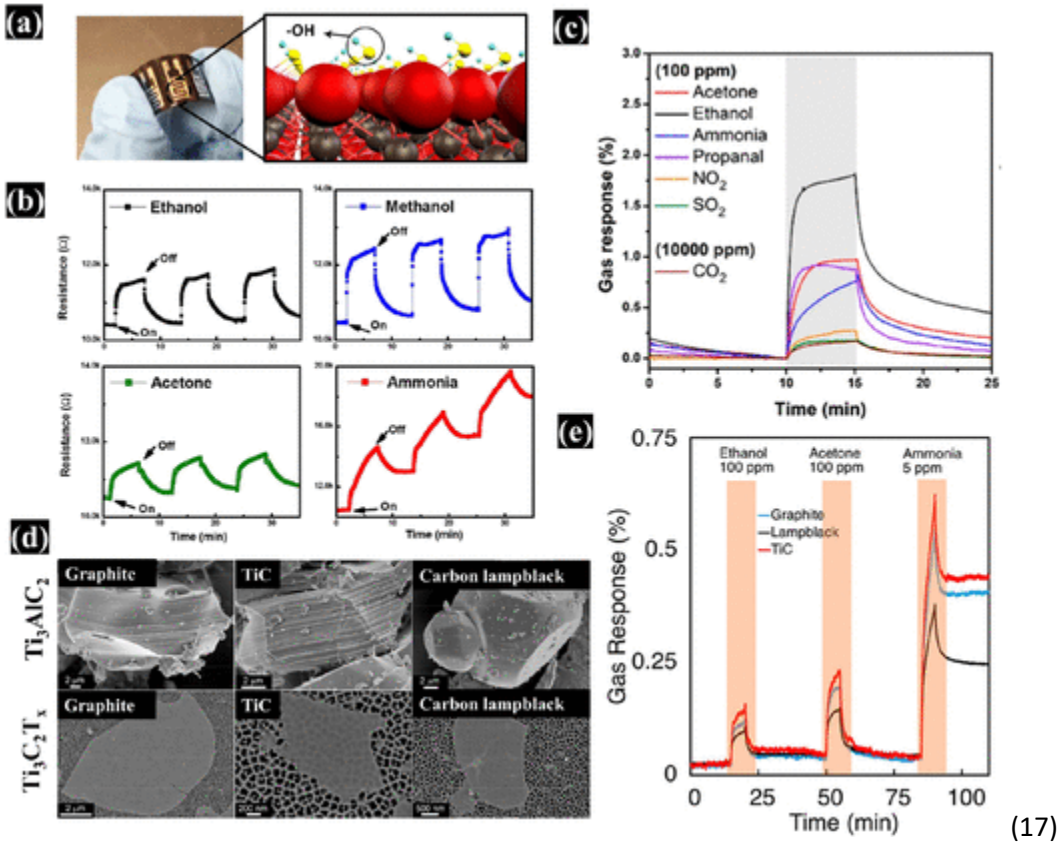
Many different MXenes including Ti_3C_2, Ti_2C, Ta_4C_3, TiNbC, V_2C, Nb_2C, Mo_2C, and Ti_4N_3 have been prepared thus far. Currently, MXene synthesis is still at the laboratory stage. The lack of a large-scale industrial production technology substantially limits their practical applications. Therefore, improving MXene synthesis methods has become a critical research topic. MXene is a layered material that is obtained by selectively removing the A phase in MAX, affording an accordion-like multilayer structure . In the resulting MXene, which is usually called multilayered MXene, the layers are mainly connected by van der Waals forces. Although the most commonly used methods for the synthesis of graphene, transition metal disulfides, transition metal oxides, and hexagonal boron nitrides are mechanical exfoliation methods most of the synthesis

methods for 2D MXenes are ***top*–down wet chemical etching methods** such as hydrofluoric acid (HF), hydrochloric acid (HCl), and lithium fluoride (LiF) mixed solution etching and fluoride-free etching. (16)

- Different methods are used to synthesis the MXene i.e. **Wet chemical etching method, Non-wet chemical etching method,etc.**

Following the successful synthesis of 2D titanium carbide MXene, its gas-sensing applications have been investigated. Lee et al. reported the first demonstration of MXene as a gas sensor by evaluating the gas-sensing properties of drop-casted $Ti_3C_2T_x$ on Pt-interdigitated electrodes fabricated on a flexible polyimide substrate under ambient atmosphere (Figure a). They observed the random functionalization of the surface of the MXene with different terminal groups like −O, −OH, and −F, with the first two dominating (Figure a). The sensor indicated p-type semiconductor behavior and its resistance enhanced after adsorption of ammonia, acetone, methanol, and ethanol gases (Figure b). The electron transfer from the gas molecules to the dominant surface terminal groups reduces the MXene film's majority carrier (holes) by electron–hole recombination, increasing the resistance. The lowest response was achieved for acetone, while the sensor presented its highest response to ammonia with a theoretical limit of detection (LoD) of 9.27 ppm. Kim et al. further investigated the volatile organic compound (VOCs) sensing performance of $Ti_3C_2T_x$ by fabrication of a metallic sensor with an exceptionally high signal-to-noise ratio (SNR). The sensor was exposed to acetone, ethanol, ammonia, nitrogen dioxide, sulfur dioxide, and carbon dioxide, while its electrical resistance was monitored (Figure c). Due to metallic conductivity and full termination of the surface with −OH and −O functional groups, the sensitivity of the sensor exceeded the conventional sensors with semiconductor channel materials, offering a LoD of 50–100 ppb for VOCs at room temperature. Regardless of being oxidative or reductive,

all the gases caused a resistance enhancement after exposure, ascribed to the metallic conductivity of the channel material in which the gas molecule adsorption limits its charge carrier transport. Additionally, the SNR of $Ti_3C_2T_x$ sensor was 2 orders of magnitude larger than those reported for other 2D materials, which is highly desirable for gas-sensing utilizations. (17)



(17)

5. Importance of study/society application:

"MXene is one of the most sensitive gas sensors ever reported. This research is significant because it expands the range for detection of common gases allowing us to detect very low concentrations that we were not able to detect before," said Yury Gogotsi.

By doing the research on MXene based Gas sensors the high sensitivity device can be made which is to be used for detecting toxic gases or pollutants found in our environment. Which will be helpful to reduce air pollution.

- **International/ National status**

  Researchers across the world working on sensors are shifting their focus to novel two dimensional materials like MXenes which can show superior performance over conventional materials.

  Among various MXene materials, $Ti_3C_2T_x$ is the most widely used and mature material, which is also the first MXene material used in gas sensors in 2017. Lee et al. used a solution casting method to integrate $Ti_3C_2T_x$ MXene (prepared by HCl + LiF etching) on a flexible polyimide platform in order to make a gas sensor. The gas response to ethanol, methanol, acetone, and NH_3 at room temperature was studied. The study showed that the resistance of the MXene film increased after the introduction of ethanol, methanol, acetone, and NH_3. (18)

  MXenes have been explored as well designed solid-state gas sensors to detect various gases with low electrical noise, high signal intensity as well as high substrate flexibility and portability. (19)

- The following scientists are actively doing research in this field at international/National level:

Yury Gogotsi's Nanomaterials Research Group, from Drexel's Department of Materials Science and Engineering, teamed with Hee-Tae Jung, PhD, a professor at KAIST in Daejeon, South Korea to explore the gas-sensing properties of titanium carbide MXene.

- **Significance of the study:**

MXenes have amazing research interest within a short period after their discovery due to their exciting layered structure and metallic-like conductivity. Researchers across the world working on sensors are shifting their focus to novel twodimensional materials like MXenes which can show superior performance over conventional materials. The good conductivity and high stability of MXenes make them an ideal candidate for the fabrication of sensors that can be employed for diagnostic applications. On the other hand, this area still needs urgent attention for making a new category of MXenes other than Titanium-based materials. Even though theoretical studies are increasing the choice of preparing MXene-based sensors from other transition metals, most of the work in this area is not successfully explored other varieties. There is a high probability and scope of developing a new category of MXene candidates which might possess good performance over the reported materials. (20)

Major challenge of present era :

''Clean energy and safe environment''

- Burning of fossil fuels causes release of hazardous gases to the environment.
- Proper monitoring of the emission of those gases is also critical.

Solution to these problems :

Ensure clean air by monitoring frequently.

Gas sensors are used for this purpose ensuring sustainability.

Gas sensor, as one of the most important devices to detect noxious gases, provides a vital way to monitor the concentration and environmental information of gas in order to guarantee the safety of production. Therefore, researches on high sensitivity, high

selectivity, and high stability has become hot issue.

The present study is devoted to syntheses a MXene material and understand the gas sensing capabilities of that material (i.e. Sensitivity, selectivity and stability etc.)

6. Proposed work plan/Formulation and structure of study:

1. First Year:

(i) Literature survey on MXene based Gas sensors and related to the topic

(ii) Coursework as per university syllabus,

(iii) Literature Review system to be studied.

2. **Second Year:**
   Syntheses MXene material ,understand the gas sensing capabilities and determine the parameters such as selectivity , sensitivity, stability and signal to noise ratio of that material.
3. **Third year:**

   (i) Paper presentation in the International/National Conference.

   (ii) Writing research Paper and publish paper in the International Journals.

   (iv) Thesis Writing and submission.

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